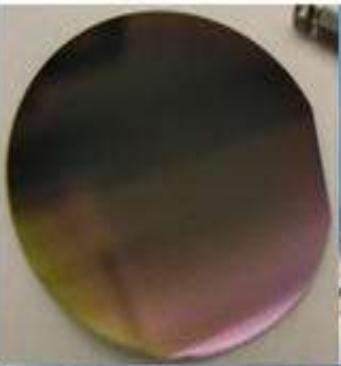




# Multi-functional Extreme Environment Surfaces: Nanotribology for Air and Space. MURI PI: J. Krim, NCSU

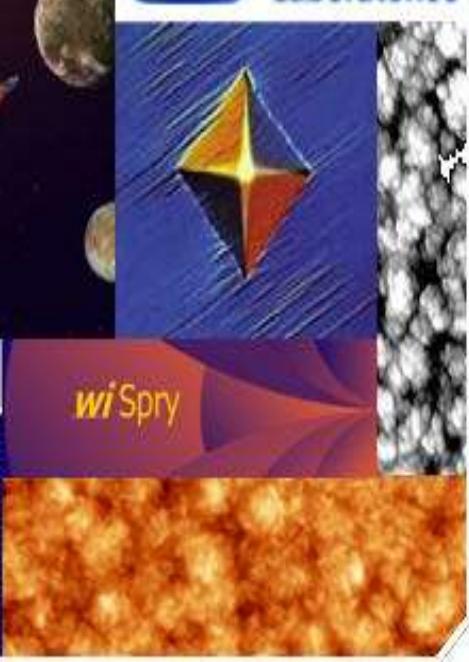
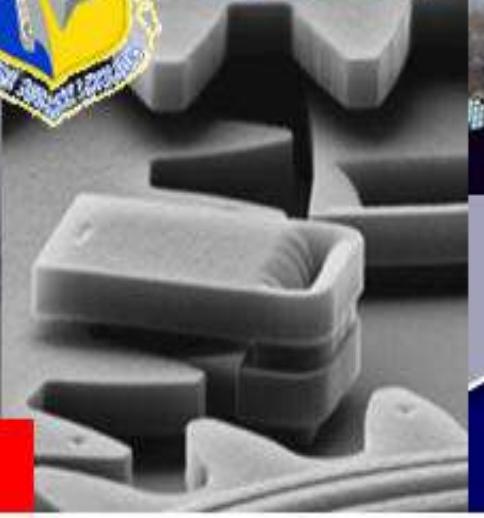


## Final Report Presentation



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Report Documentation Page		Form Approved OMB No. 0704-0188
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14. ABSTRACT <p><b>We have: *Discovered that ethanol extends silicon MEMS lifetimes at ultra-low coverages, and proposed fundamental mechanisms for the exceptional lubricating properties. *Derived a new multi-scale modeling code for continuum heat and current flow in nanowires at de-adhering gold-gold contacts. *Applied our finite-element algorithms techniques to investigate nanocomposites with grains down to 20 nm over a broad spectrum of extreme loading conditions. *Developed new models of surface roughness and materials properties for RF MEMS contact and the transition from plastic deformation to creep behavior. *Developed a classical potential energy function capable of accurately modeling chemical reactions in carbon, hydrogen, and oxygen containing materials, allowing first time studies of DiamondLikeCarbon (DLC) in the presence of water. *Discovered that the run-in friction behavior of NanoCrystallineDiamond coatings is strongly correlated with the structure of sp2 carbon in *Performed an STM study of boron-doped diamond films that revealed growth features possibly due to quantum nanoscale ?magic-sized effects.</b></p>		
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# Multi-functional Extreme Environment Surfaces: Nanotribology for Air and Space. MURI PI: J. Krim, NCSU



NC STATE UNIVERSITY

## Academic Team Members

**Jacqueline Krim**,(a) Prof. of Physics & Assoc. of ECE

**Donald W. Brenner**,(a) Prof. of Materials Science & Eng. (MS&E)

**Judith A. Harrison**,(b) Prof. of Chemistry

**Angus I. Kingon**,(h) Prof. of Materials Science & Eng

**James Rutledge**,(c) Prof. of Physics

**Peter Taborek**, (c) Prof. of Physics

**Mohammed A. Zikry**,(a) Prof. of Mechanical & Aerospace Eng.

## Specialty

Nanotribology

Computational Triboochemistry

Computational Nanotribology

Nanomaterials Design

Cryotribology

Cryotribology & Coatings

Computational Nanocomposites

## DoD & DOE Team Members

**Michael T. Dugger**,(d) Ph.D, Material Science and Engineering

**Kathryn Wahl**,(e) Ph.D, Material Science and Engineering

**Andrey A. Voevodin** (f) Ph.D, Chemical Engineering

## Specialty

Microtribology & MEMS

Tribocoating Analyses

Aerospace Tribocoatings

## Industrial Partner

**Art S. Morris III**,(g) Ph.D, wiSpry Inc.

## Specialty

RF MEMS

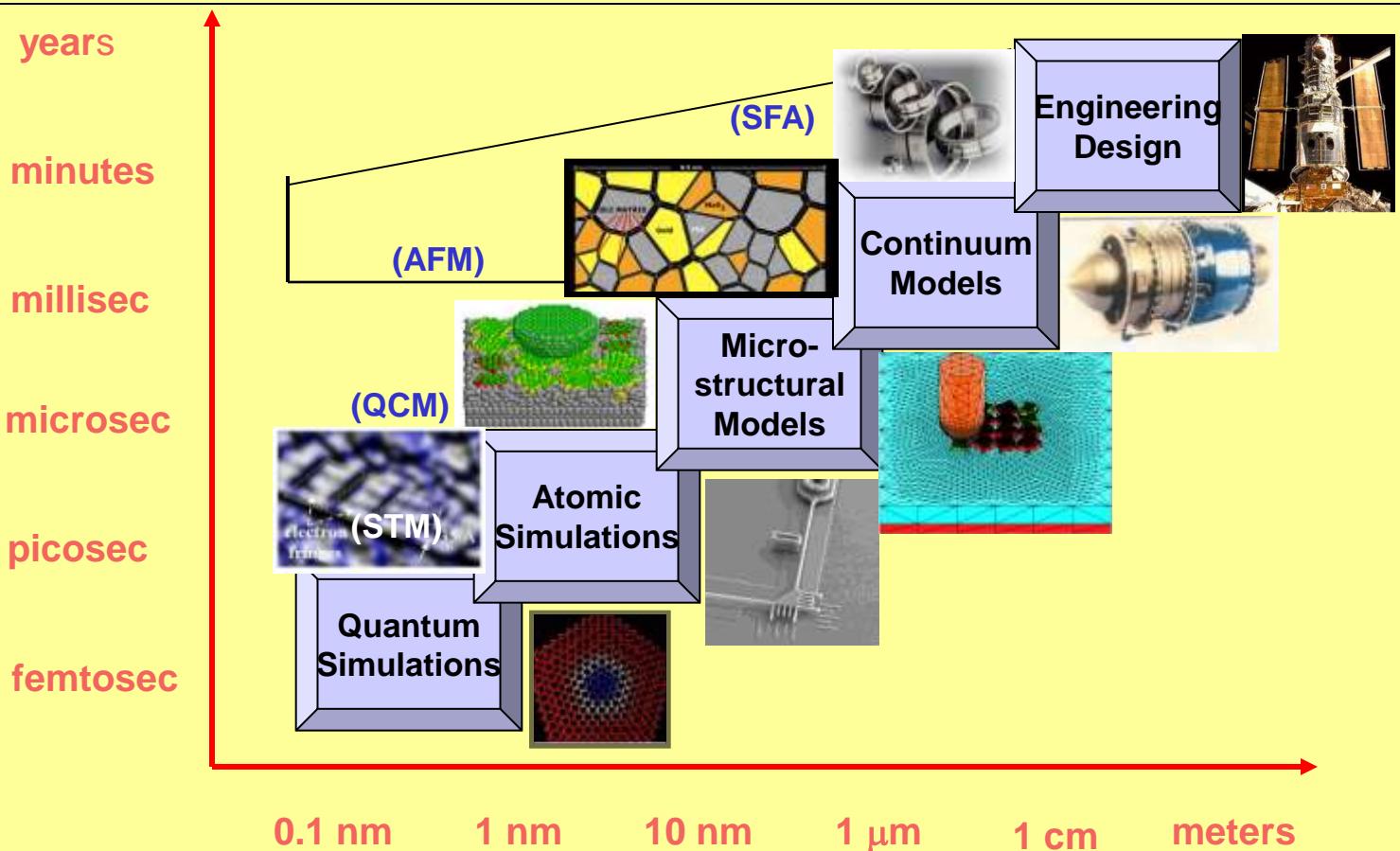
**Participating Institutions:** (a)North Carolina State University, (b)United States Naval Academy, (c)University of California-Irvine, (d)Sandia National Laboratories, (e)Naval Research Laboratory, (f)Wright Patterson Air Force Research Laboratory, (g) wiSpry Corp.(h)Brown University



# Multi-functional Extreme Environment Surfaces: Nanotribology for Air and Space. MURI PI: J. Krim, NCSU



## SPANNING THE PHYSICAL SCALES OF MODERN TRIBOLOGY



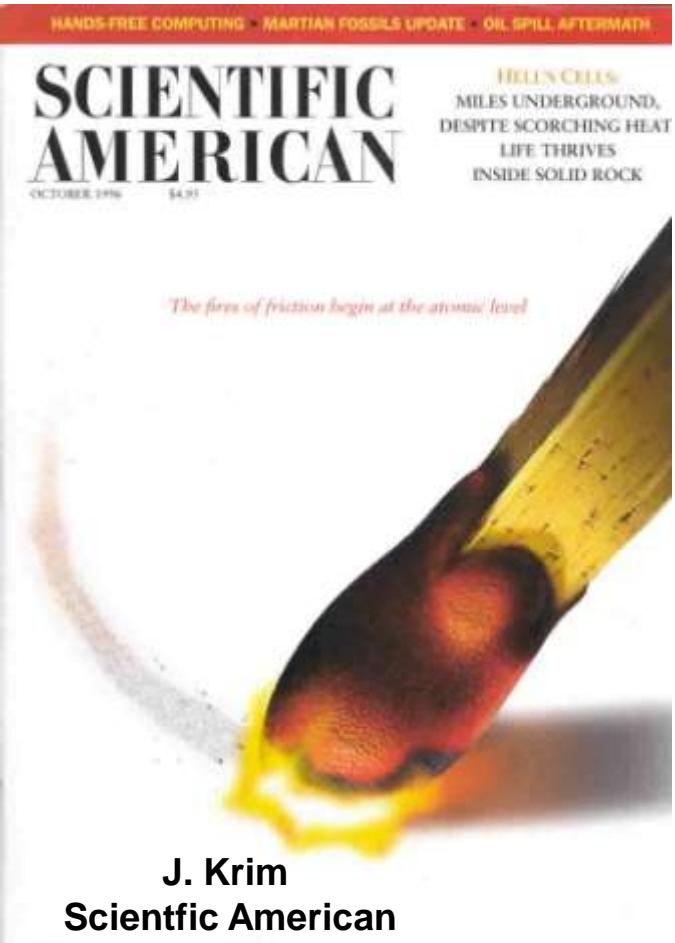
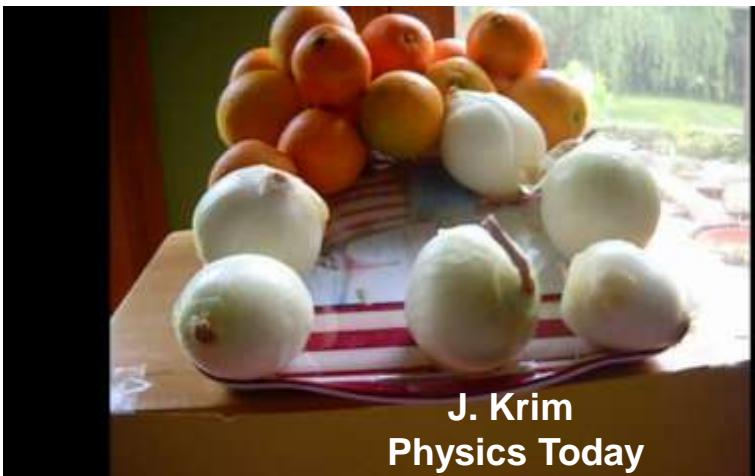


# Fundamental Challenges and Unsolved Issues

*How do adsorbed and tribo-generated films impact friction and wear?*

*How is heat dissipated in a system and what temperature rise is associated with it?*

*How do electrons impact friction?*

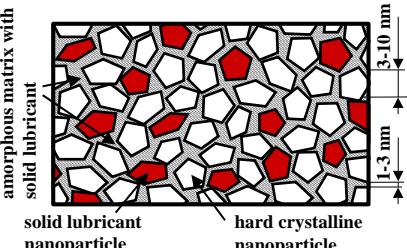
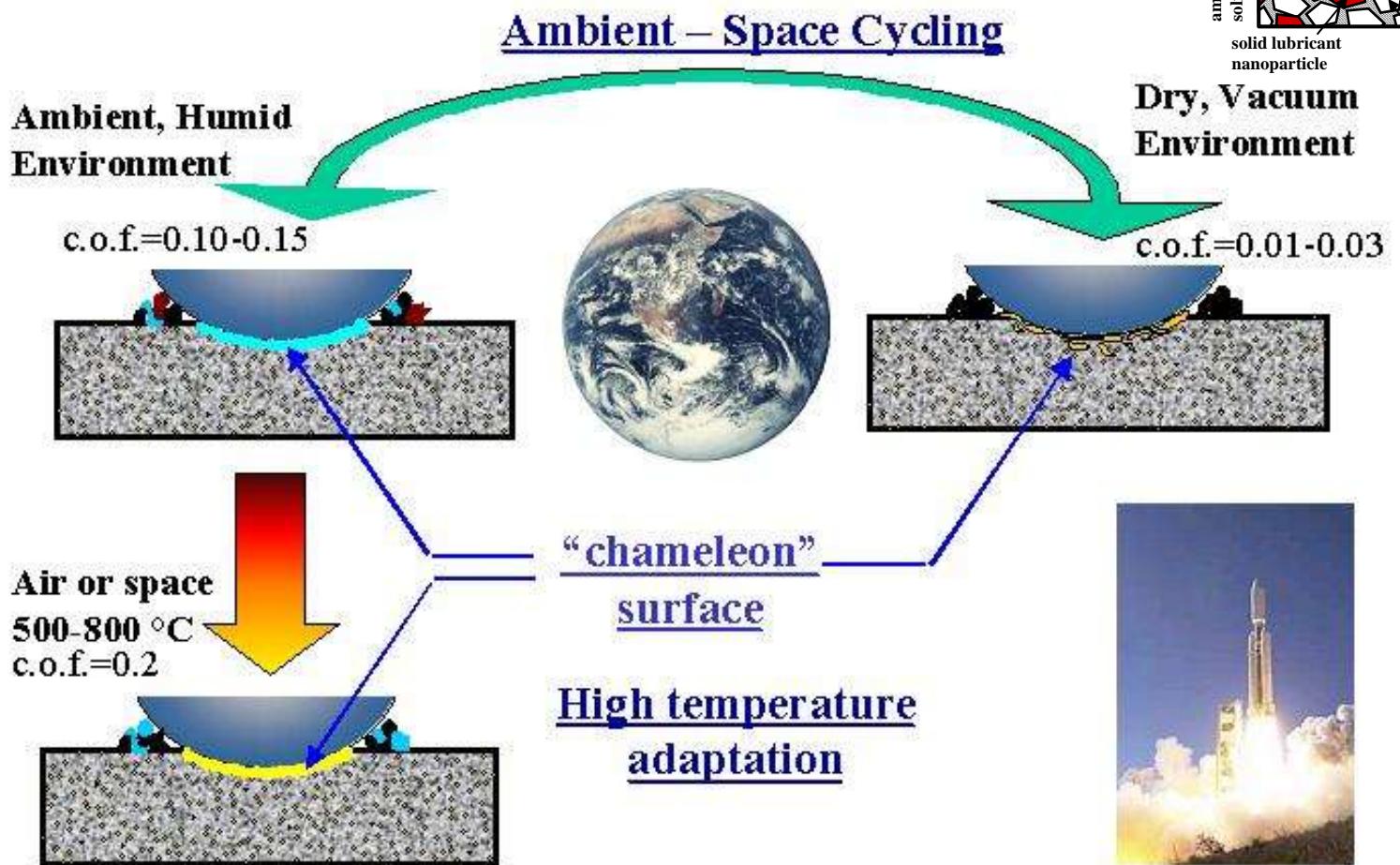




# Thrust I: Nanocomposite Coatings for Terrestrial Applications



*Gears and bearings for aircraft and jet engines;  
Reusable launch vehicles*

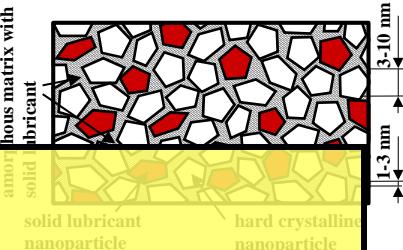




# Thrust I: Nanocomposite Coatings for Terrestrial Applications



*Gears and bearings for aircraft and jet engines;  
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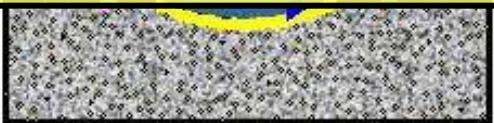
**Five Years ago:**

Ambient, Humid  
Environment  
 $c.o.f = 0.10 - 0.15$

Dry, Vacuum  
Environment  
 $c.o.f = 0.01 - 0.03$

**Speculation on the roles of constituents;  
Edisonian approach.**

**Today:**  
**Combined multiscale modeling-experimental collaboration allow us to predict tribological performance from film nanostructure.**



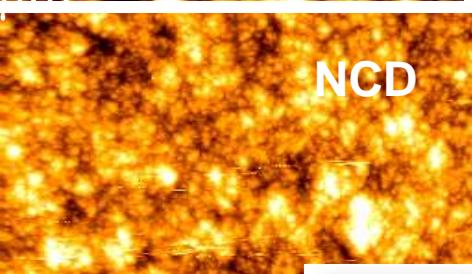
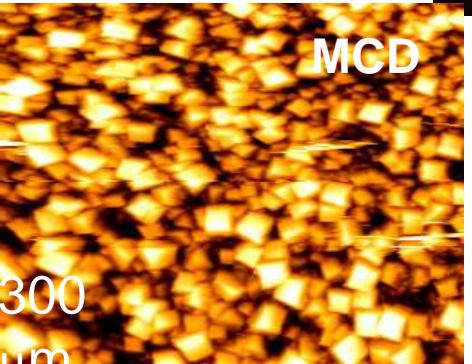
adaptation





# Thrust II: Cryotribology and Nanocrystalline Diamond for Space Applications

*Satellite bearings, InfraRed sensor mechanisms  
Jet engine bearings*





## Thrust II: Cryotribology and Nanocrystalline Diamond for Space Applications



*Satellite bearings, InfraRed sensor mechanisms*

*Jet engine bearings*

**Five Years ago:**

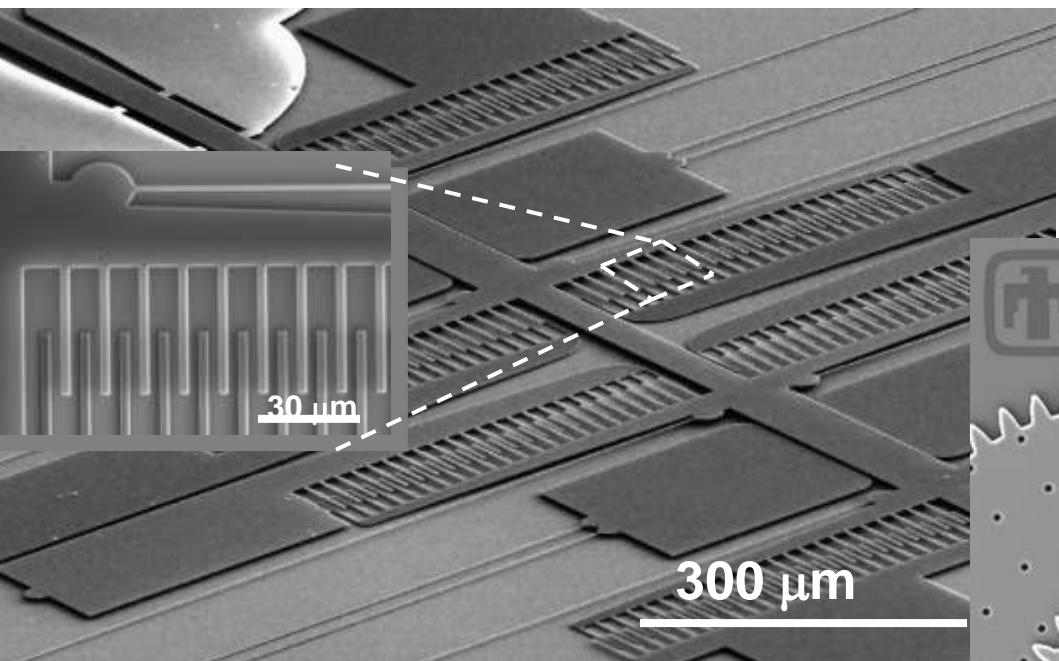
**Three publications in the area of vacuum cryotribology; reactive water potential nonexistent**

**Today:**

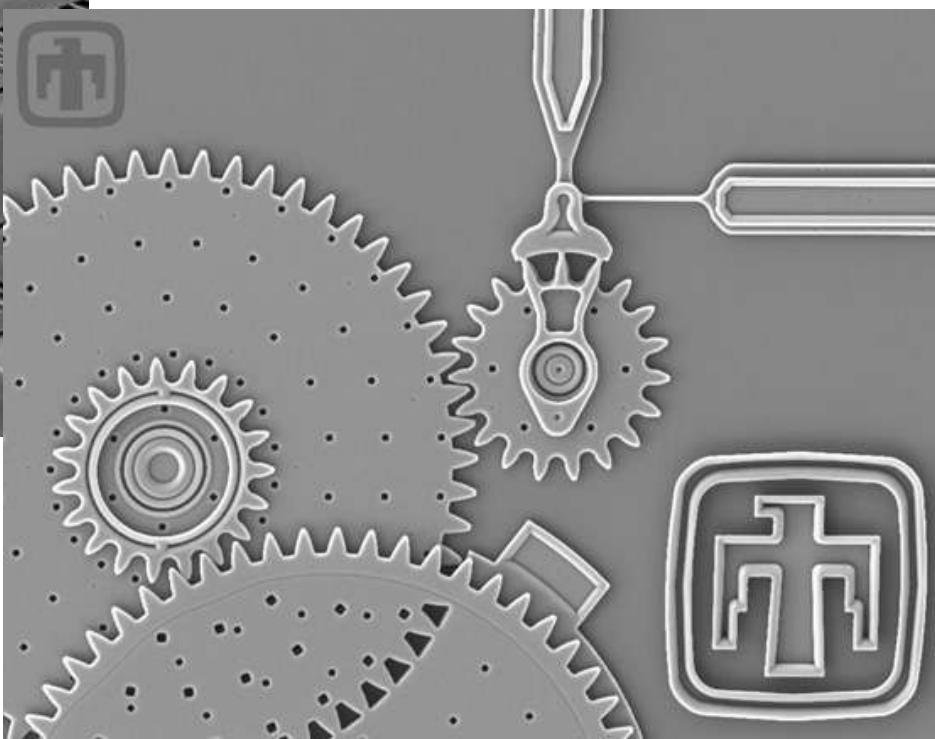
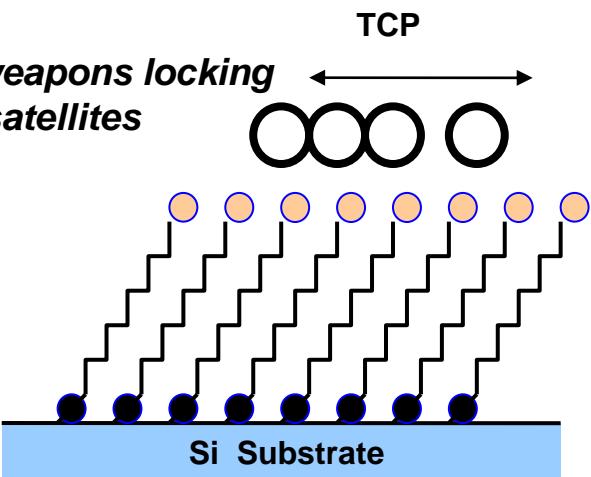
**An emerging and lively field of cryotribology with interplay of experimental data and fundamental theory. A viable reactive code for water has been completed.**



## Thrust III: Silicon MEMS ; Bound + Mobile Lubrication



*Communications, electronics, sensors, weapons locking  
miniature air vehicles, miniature satellites*





TCP

*Communication electronics, sensors, weapons locking  
miniature air vehicles, miniature satellites*

### Five Years ago:

**Silicon MEMS devices with rubbing contacts degraded within minutes. Focus on SAMS techniques to prevent tribological failure.**

**Today:**

**SAMS ruled out for protection against device failure in rubbing contact. Devices operational for indefinite durations in the presence of trace levels of alcohols. Atomic scale mechanisms proposed for multiple lubrication schemes.**

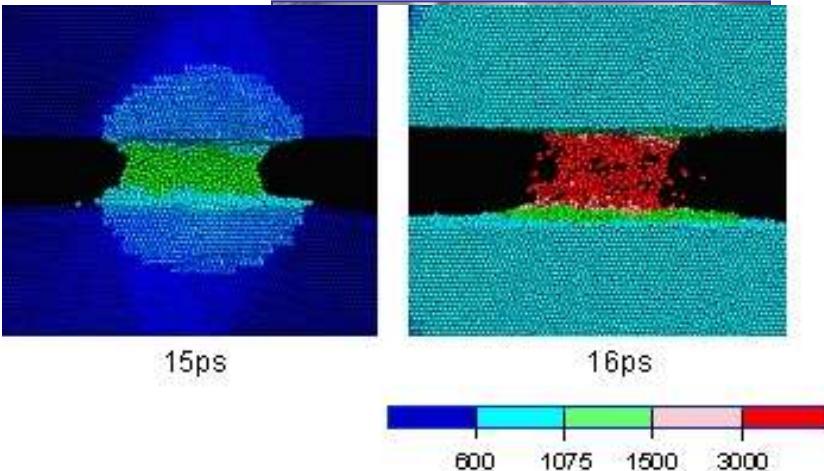
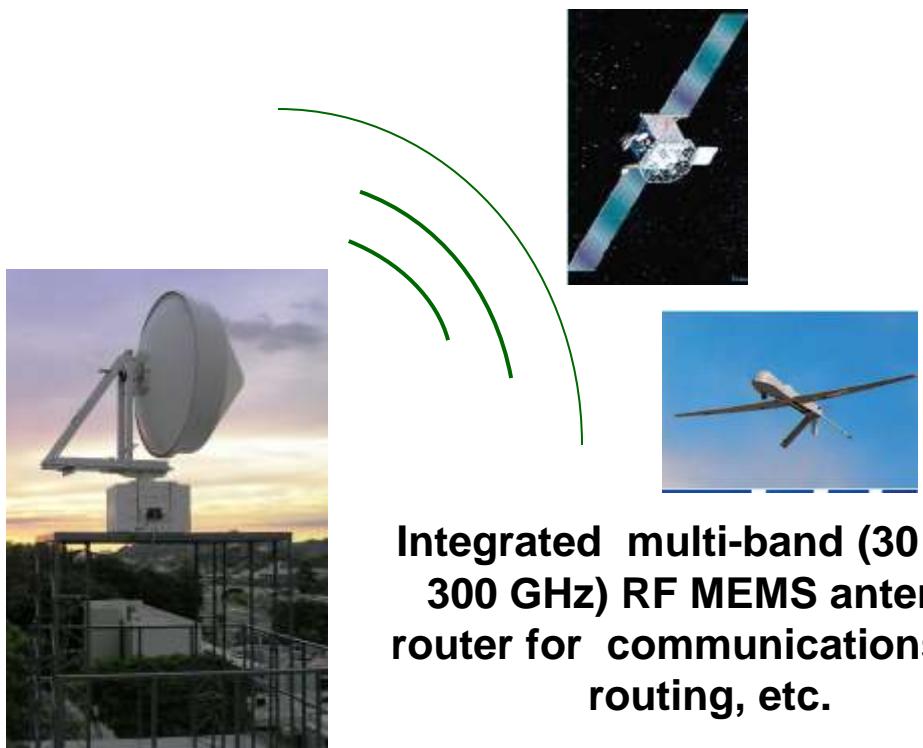


# Thrust III: RF MEMS Applications

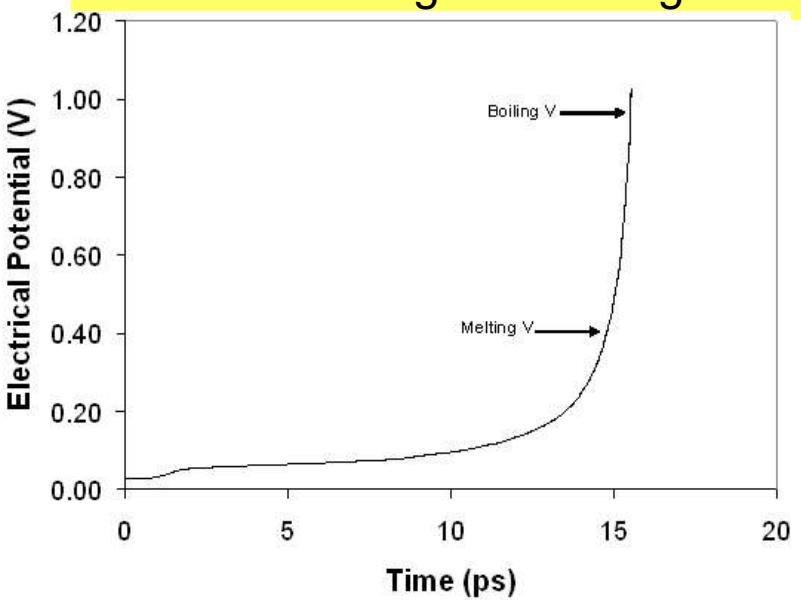
Compared to conventional devices, RF-MEMS have:

- ✓ Small size
- ✓ Low power consumption
- ✓ Wide band width
- ✓ Good signal discretion

**Portable & Versatile**



Snapshots from simulations of wire melting and boiling





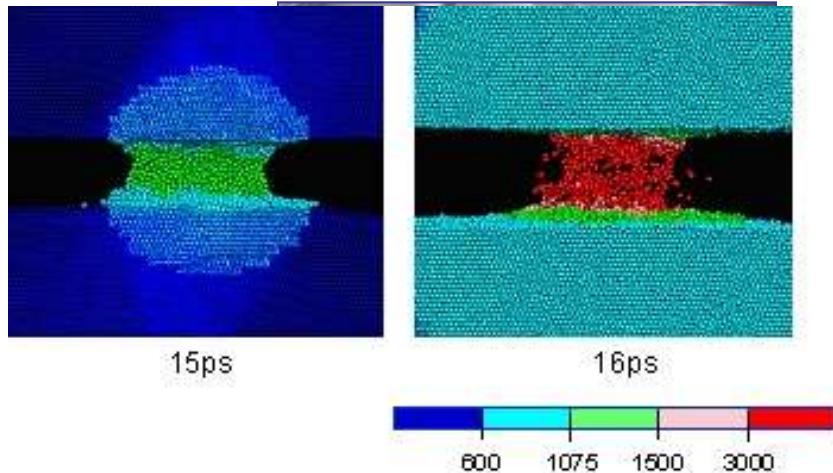
# Thrust III: RF MEMS Applications

# Molecular Simulations of Hot Switching devices, RF of Gold Contacts

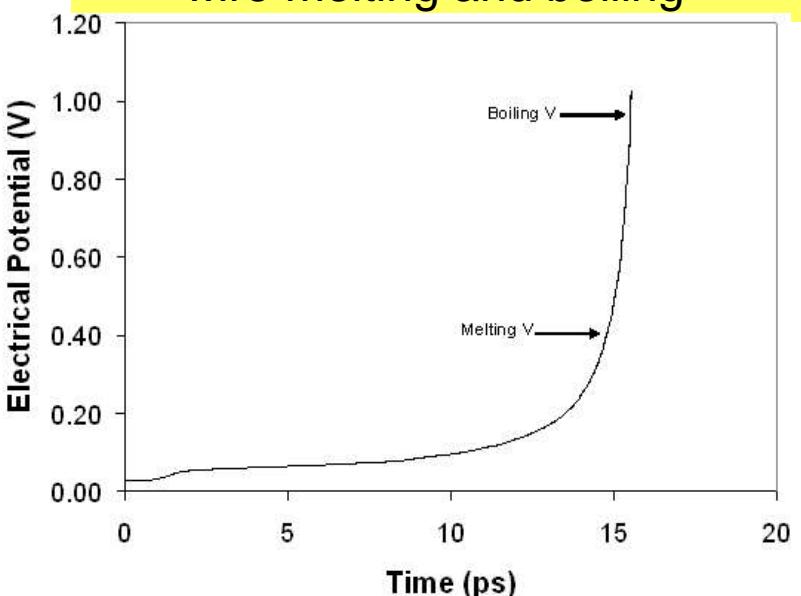
- Atomic simulations demonstrated nano-wire formation with diameter much smaller than initial contact area
  - Consistent w/experiments on MEMS, other gold contacts
  - First modeling to reveal nanowires form from larger contact areas (made possible by collaboration with Zikry)
    - For cold switching, wire forms when dislocations from step edges are pinned
    - Hot switching enhances dislocation mobility, leads to melting and vaporization of contact.
    - Scaling wire size + boiling yields upper bound for material transferred that agrees with Kingon result.

**Current studies:**

- Modeling other contact orientations to see if material transfer scaling is orientation dependent.
- Developing thermal ionization model to quantify material transfer



# Snapshots from simulations of wire melting and boiling





# Publications, Presentations

>100 referred publications in refereed journals, book chapters, cover stories, encyclopedia articles and conference proceedings.

(2005) National Space and Missile Materials Symposium, Outstanding Poster (1st Prize), Operating in Space Session, *Douglas L. Irving et al.*

>70 invited talks, including 4 keynote/plenary lectures at conferences.

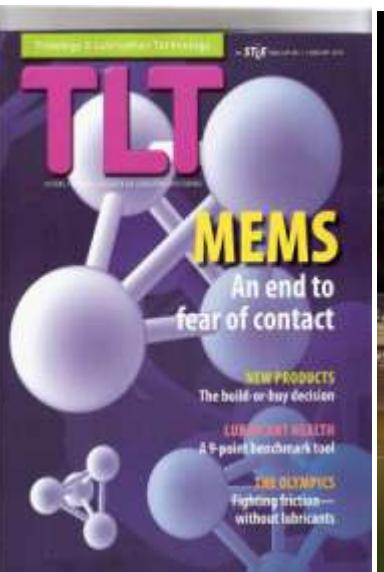
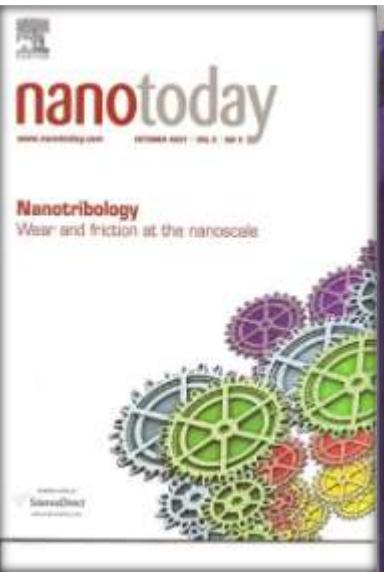
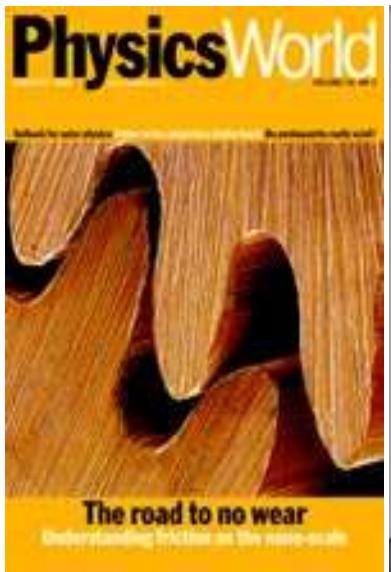
(2005) World Tribology Congress, Washington DC: *Harrison* , Keynote lecture

(2005) University of Columbia, MO: *Brenner*, Plenary lecture

(2009) ViennaNano , Vienna, Austria: *Krim*, Plenary lecture

(2009) Advances in Boundary lubrication, Seville, Spain : *Krim, Harrison invited keynote lectures*

Cover stories by *Wahl, Dugger, Krim*





# Honors & Awards

- 2004-2005: *M. A. Zikry, Senior Fulbright Research Award*
- 2006: *K. Wahl, AVS Fellow, For exceptional contributions to the fundamental understanding of contact mechanics, adhesion and tribology at the nanometer and micron scales*
- 2006: *A. Kingon, Price Foundation Award , Innovative Entrepreneurship Educator*
- 2008: *M.A. Zikry, Jefferson Science Fellow , US State Department (2008), initiated by the Science and Technology Adviser to the Secretary of State to further build capacity for science, technology and engineering expertise within the Department.*
- 2008: *D. E. Brenner, Appointed to Kobe Steel Distinguished Professorship in NCSU Materials Science and Engineering Dept.*
- 2008: *A. Kingon, Barrett Hazeltine University Professor of Entrepreneurship , Distinguished Professorship at Brown University (July 2008)*
- 2008: *K. Wahl, Department of the Navy - Meritorious Civilian Service Award, for Exceptional contributions to the advancement of Naval Research in the fields of contact mechanics and the chemistry of adhesion, friction and wear.*
- 2009: *J. Harrison, recipient of the George L. Braude, Award, presented by the Maryland Section of the American Chemical Society to “a chemist with significant standing and committed to supporting student research.”*
- 2009: *D.E. Brenner, RJ Reynolds award for research, teaching and extension.*
- 2010: *J. Krim, NSF American Competitiveness and Innovation (ACI) Fellow, “For outstanding contribution to understanding friction at the nanoscale and exemplary efforts in broadening participation in science through maintaining a diverse research group and through explaining her research to the lay public.*



# Student Development



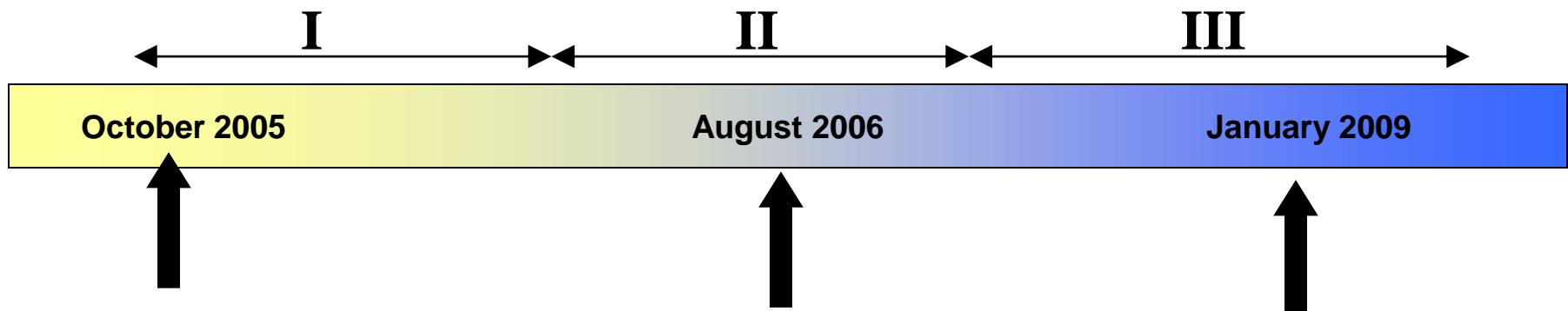
- **Group Alumni:** are presently placed as staff scientists, analysts and process engineers throughout the US and Canada.  
**Government labs:** NRL, Army, USDA, ARL  
**Industry:** Cree, Applied Materials, Inc, Boeing Corp., Linear Technology, Inc, Northrop Grumman  
**Academia:** faculty members at McGill University, NC State, post-docs at U of Wisconsin , U of Pennsylvania, and Washington State U.  
**Continuing studies:** Harvard Business MBA, NCSU Nuclear Engineering Ph.D.
- At the project completion point, **more than 50 students and post-docs** will have been trained with direct experience in areas of national need, including 15 Ph.D.'s.



# Project Schedule and Milestones



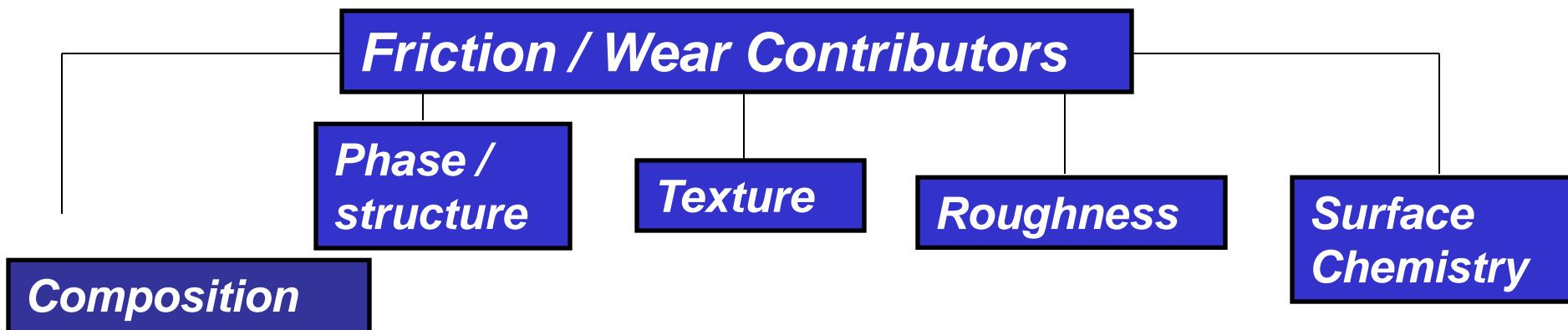
- **Phase I: Combining existing expertise.** (May 2004 – November 2005)  
In this beginning period, the separate expertise at the various partner institutions was combined and materials' standards were developed to produce baseline material.
- **Phase II: Exploitation of new capabilities** (December 2005 – April 2007)  
In this period, the combination of separate expertise at the various partner institutions was completed, and active data recording with the new tools available was performed by our research team.
- **Phase III: Deliverables, Technology transfer and Commercialization**  
(May 2007 – May 2010)





# Wahl/Zikry Goals and Objectives

- Goal: Develop *in situ* analytical methods to identify how nanostructured materials impact friction and wear performance.
- Problem Statement: Without *in situ* and real-time methods, it is nearly impossible to determine how friction evolves with sliding
- What we have learned: Tribology of nanocomposite materials is dominated by interfacial film formation and coating mechanical properties
- In nanocrystalline diamond, the presence of graphite crystallite structure is more effective than amorphous  $sp^2$  carbon in reducing run-in friction.

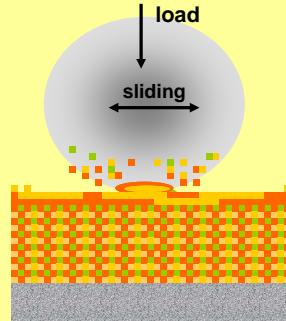




# Highlight – Adaptive Nanocomposites

## EXPERIMENTAL OBSERVATION

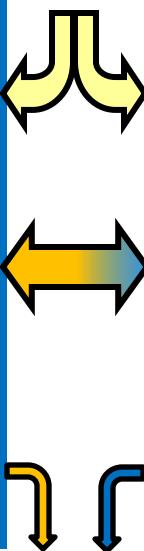
Tribology of nanocomposite materials is dominated by interfacial film formation and coating mechanical properties



## MODELING INTERFACIAL PROCESSES

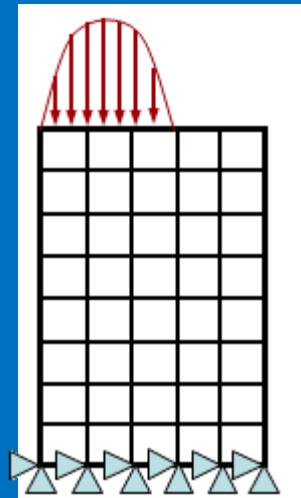
FE coating components mimic real coating constituent mechanics and scale

Failure criterion allows generation of interfacial films



## WEAR MODELING

Iterative approach allows wear simulation at realistic spatial and temporal scales



## Broader Impact

Predictive methodology for determining durability of nanocomposite coatings in terms of optimal coating material constituents and mechanical properties

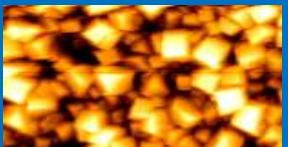


# Highlight – Nanocrystalline Diamond

## EXPERIMENTAL OBSERVATION

**Nanocrystalline diamond:** Roughness and non-diamond chemistry (graphite, CH<sub>x</sub>, ...) play a determining role in abrasiveness, run-in friction, and environmental sensitivity.

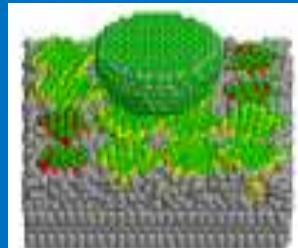
### EXPERIMENTAL



Materials science and chemistry to determine coating constituents and morphology - not deposition conditions - predict tribological performance

### MODELING & SIMULATIONS

Hierarchical MD & FE results qualitatively agree  
New potentials enable MD simulations of diamond nano-composites in presence of water and oxygen

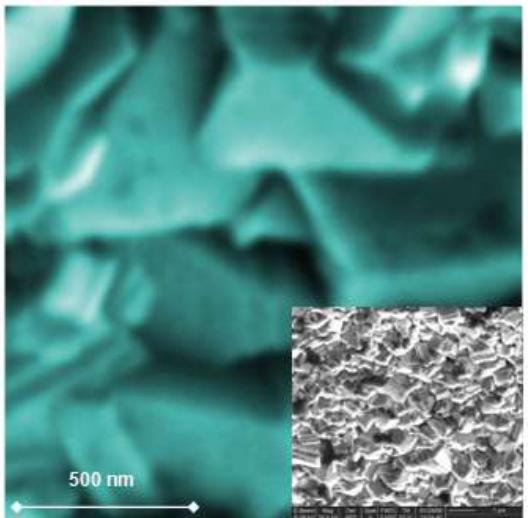


## Broader Impact

We can predict nanostructured coating tribological response and better select desired components at design stage

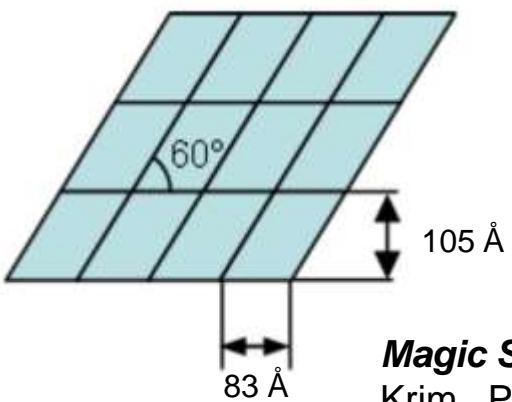
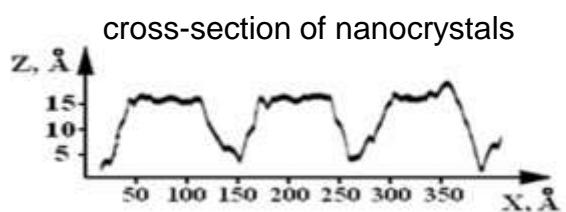
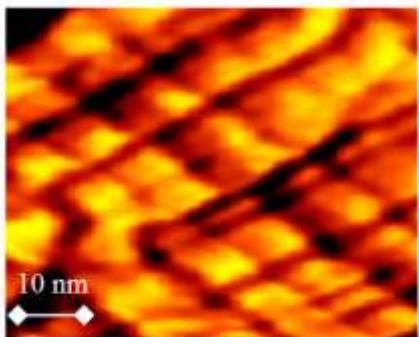
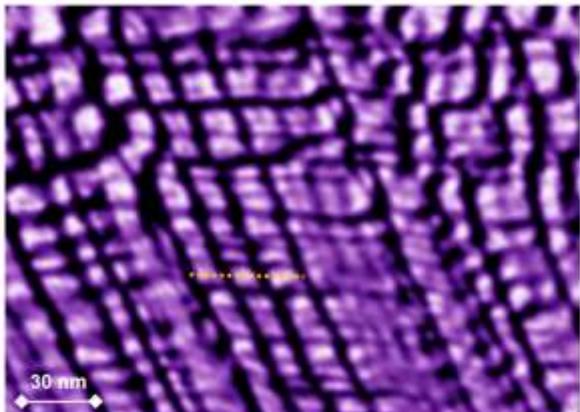


## Scanning tunneling microscopy of B-doped diamond

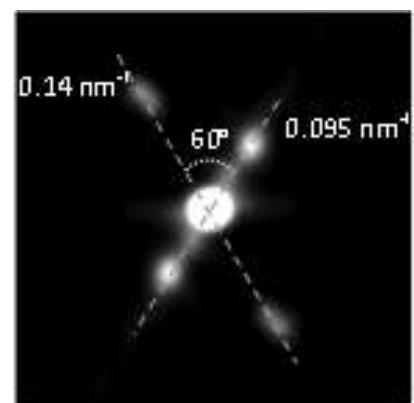


Diamond grains on large scale STM and SEM (insert) images

Arrays of magic-sized nanocrystals on top of diamond grains



The surface of B-doped diamond is comprised of spatially ordered parallelogram-shaped nanocrystals whose height ratio is 2/3



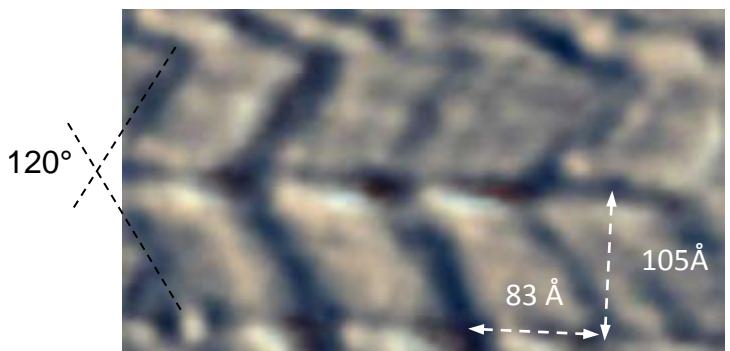
The 2D STM Fourier transform. Two 60°-rotated spatial periods whose ratio is 2/3

**Magic Sized Diamond Nanocrystals**, I. B. Altfeder, J. J. Hu, A. A. Voevodin, J. Krim, Phys. Rev. Let. **102**, 136104 (2009) ; Media coverage: Quantum Control of Diamond nanostructures, Chemical and Engineering news, April 13, 2007 vol **87**, 44

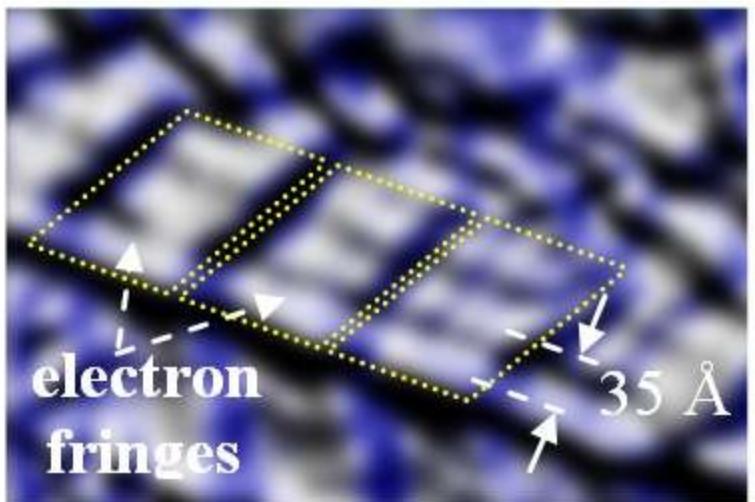


# Scanning tunneling microscopy of B-doped diamond

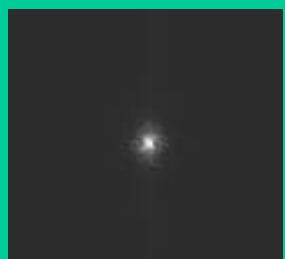
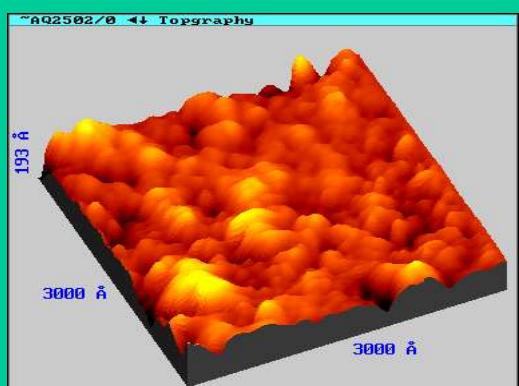
Anti-phase boundaries of nanocrystals



STM imaging of top surface on nanocrystals



Comparison with undoped diamond. Real space STM image and its 2D Fourier transform do not reveal magic sizes



At high boron densities diamond becomes metal (Nature 428, 542, 2004). The 35-Å-spacing between the lateral electronic fringes is  $\frac{1}{2}\lambda_F$

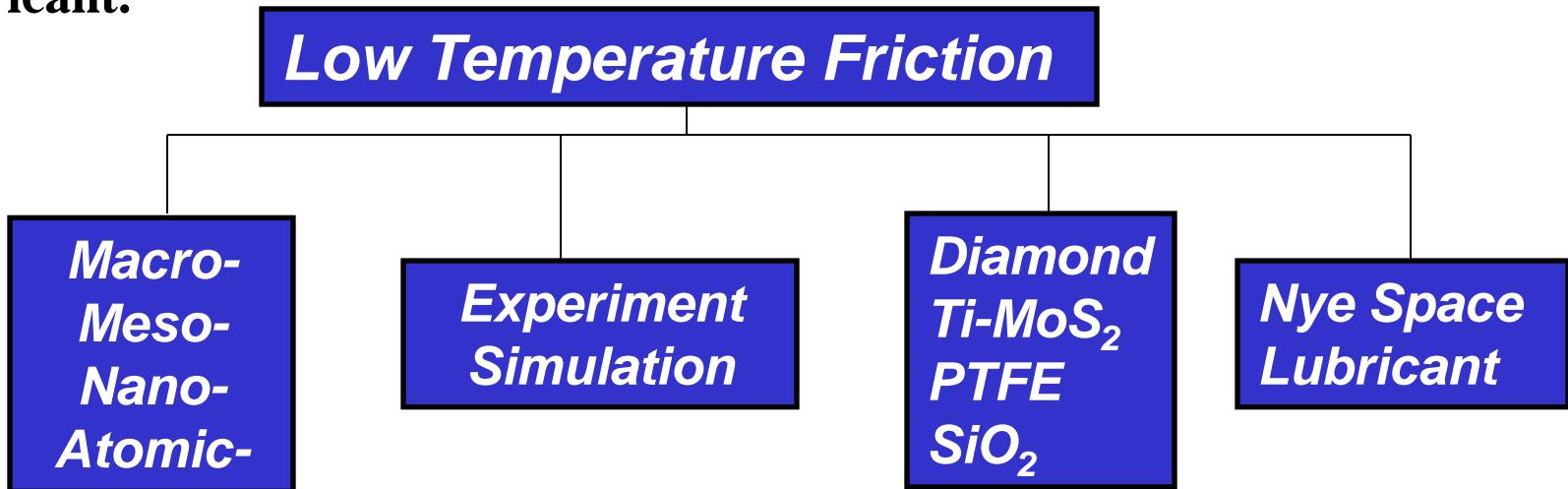
## Conclusion:

**Magic sizes in B-doped diamond are induced by electronic quantum size effect. Similar growth effects were previously observed for thin metal films and molecules adsorbed on metals.**



# Cryotribology: Goals and Objectives

- **Goals:** Identify the fundamental physical mechanisms of friction using controlled environments and cryogenic temperatures.
- **Problem Statement:** How does friction depend on temperature for a variety of materials over a range of length scales? How can we experimentally probe the role electrons and phonons in friction?
- **What we have learned:** Macroscopic dry sliding friction is the same on both sides of the superconducting /normal transition for dry contact of Nb/Nb and Stainless steel/Nb, but not ruled out for He, which is a low temperature lubricant.

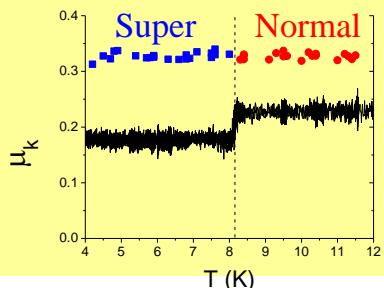




# Cryotribology

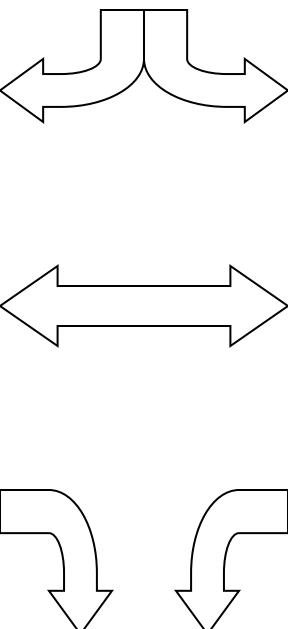
## Experimental Observation

Macroscopic friction appears independent of the superconducting or normal state of a metal for dry contact, but not helium films.



## Materials

- DLC
- YBCO
- Niobium
- Helium



## Modeling

- Phononic friction
- Heat flow
- Tomlinson hopping models
- Simulations show same trends as AFM measurements of diamond and NCD as well as Sang Theory.

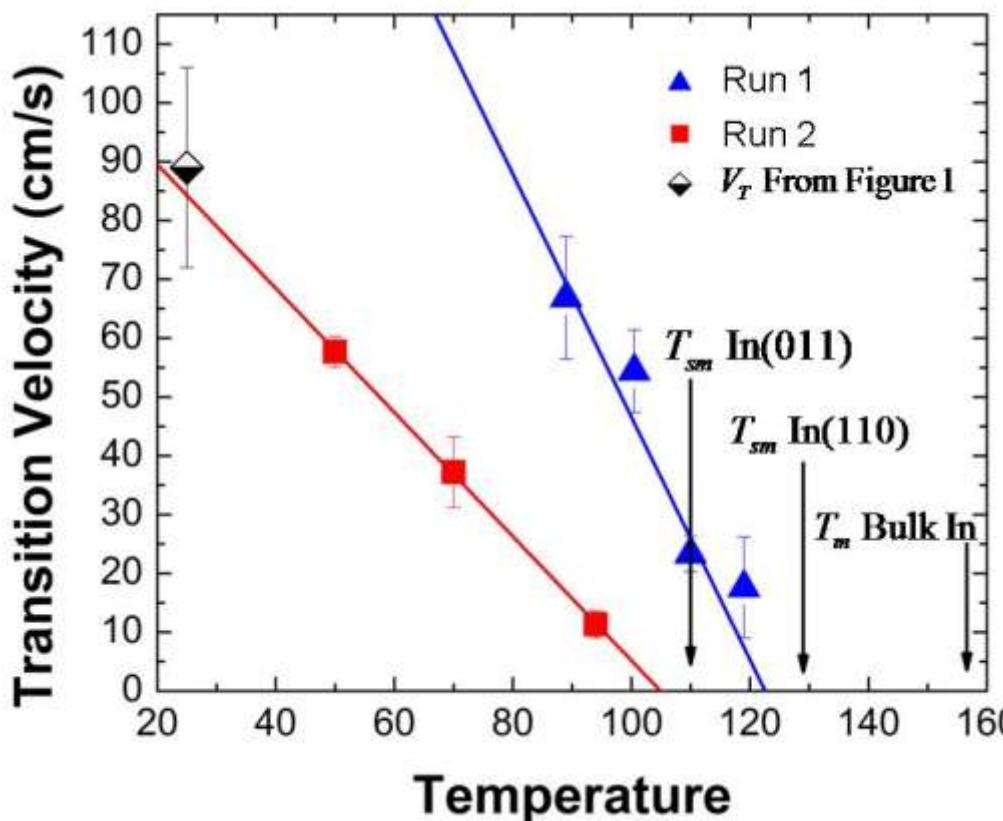
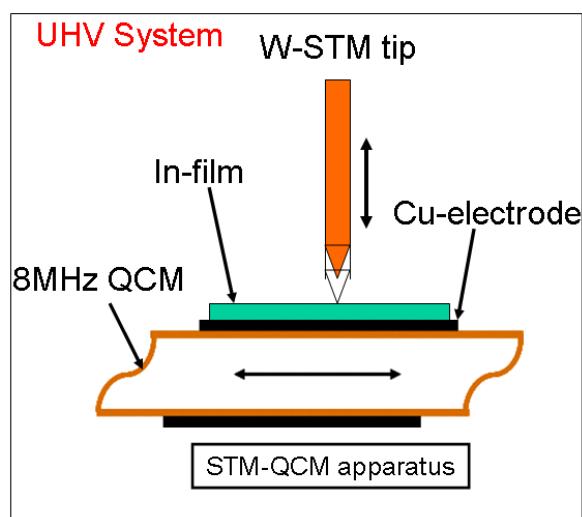
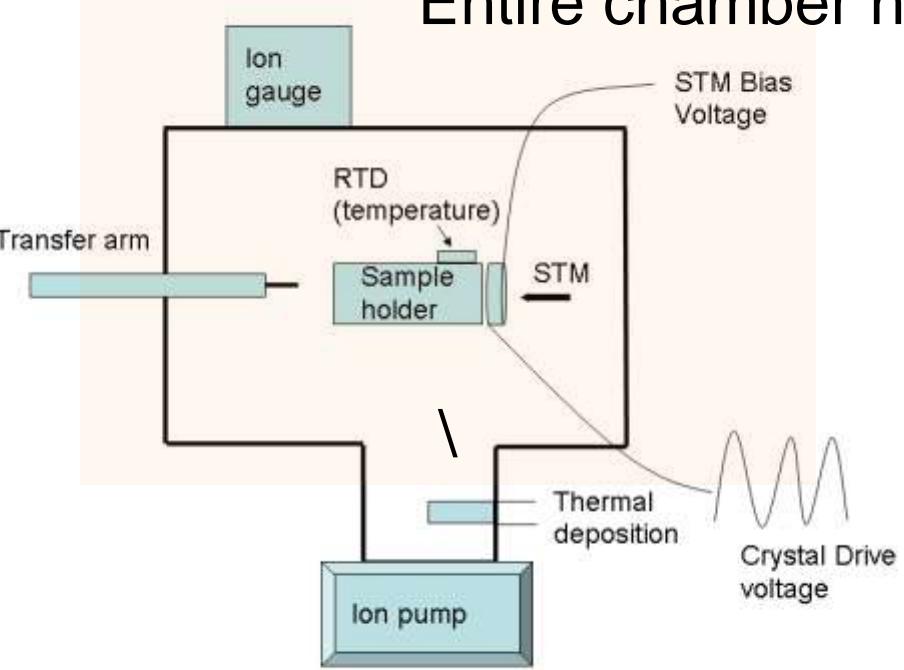
## Broader Impact

Electronic phase transitions are promising means of manipulating non contact friction.



# Tribo-induced melting at a nanoasperity

Entire chamber heated



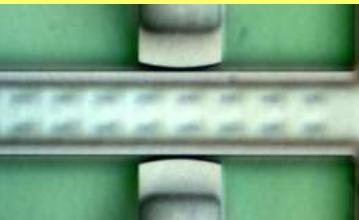
B.D. Dawson, S.M. Lee and J. Krim,  
PRL, 2009, submitted



# Si MEMS Lubrication

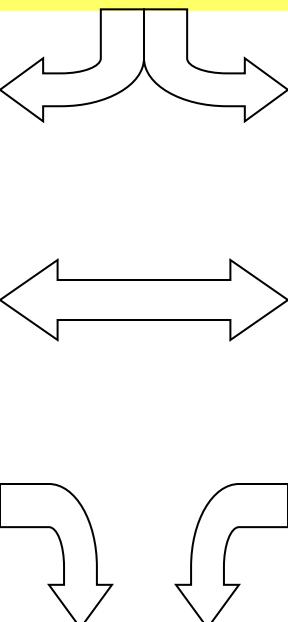
## Experimental Observation

Vapor phase lubrication solves the MEMS lubrication problem.



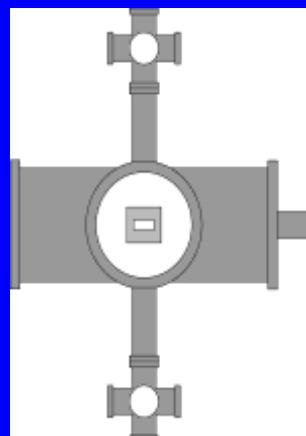
### MEMS Experiment

- MEMS tribometer confirms lubrication by 1-pentanol and shows lubrication by ethanol.
- Rigorously controlled environment allows identification of different mechanisms for these two alcohols.



### QCM Experiment

- Complementary information clarifies mechanisms
- Traditional technique for nano-scale Physics.



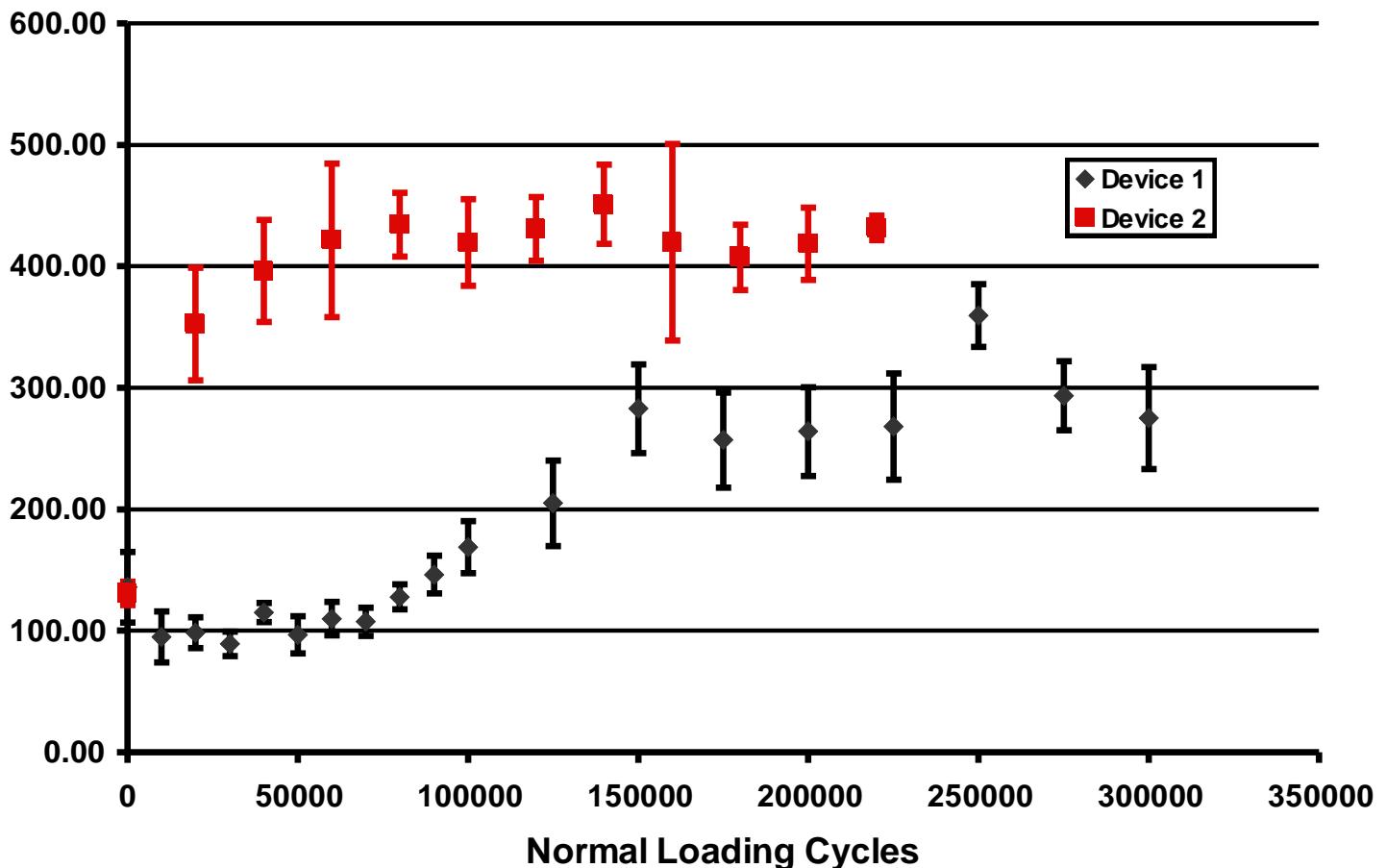
## Broader Impact

Single and submonolayer lubrication is relevant far outside the nanometer device regime.



D.A. Hook, S. Timpe, M.T. Dugger, J. Krim, J. Appl. Phys., 104 (2008)  
***“SAMS don’t work and they never will : They rub off”\****

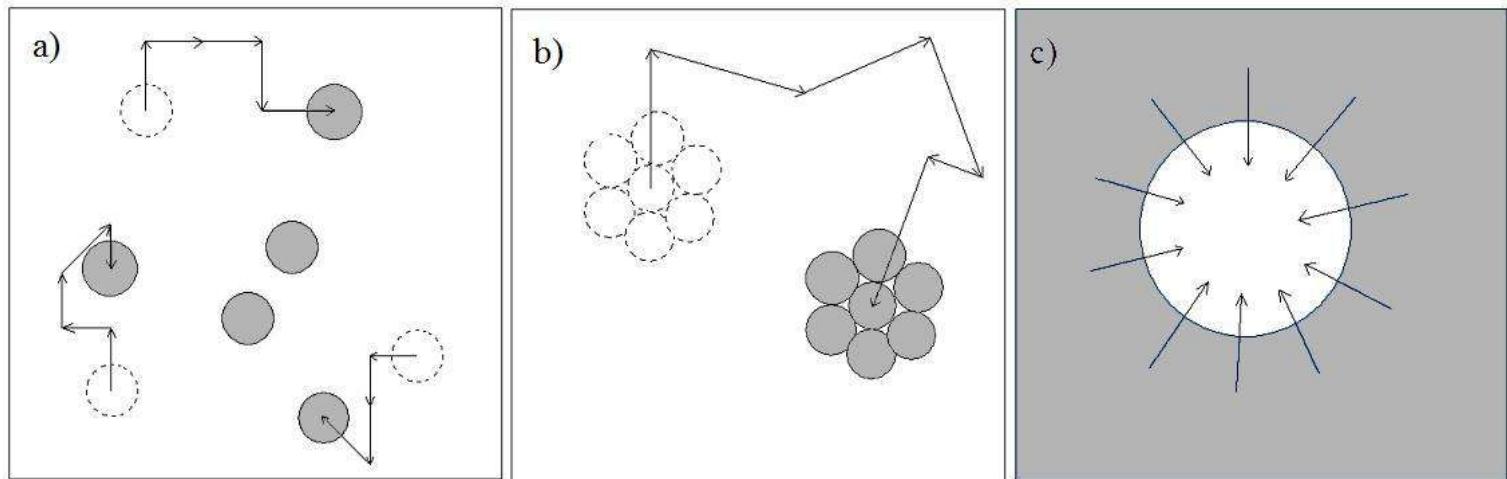
Normal Contact Degradation



\*But there is a silver lining



## Nanoscale Sliding friction and diffusion coefficients for alcohols



	PFTS			Si			Al		
	$\tau$ ns	$D_s$ cm <sup>2</sup> /s	$D_i$ cm <sup>2</sup> /s	$\tau$ ns	$D_s$ cm <sup>2</sup> /s	$D_i$ cm <sup>2</sup> /s	$\tau$ ns	$D_s$ cm <sup>2</sup> /s	$D_i$ cm <sup>2</sup> /2
Pentanol	6	1.2	$5 \times 10^{-5}$	6	1.1	$5 \times 10^{-5}$	0	0	0
Ethanol	4	1.1	$4 \times 10^{-5}$	8.5	9	$1 \times 10^{-4}$	0.5	0.1	$6 \times 10^{-6}$
TFE	0	0	0	3.5	1.3	$2 \times 10^{-5}$	0	0	0

Sufficient mobility exists at the nanoscale for ethanol and pentanol to diffuse both in the presence and absence of a SAMS layer

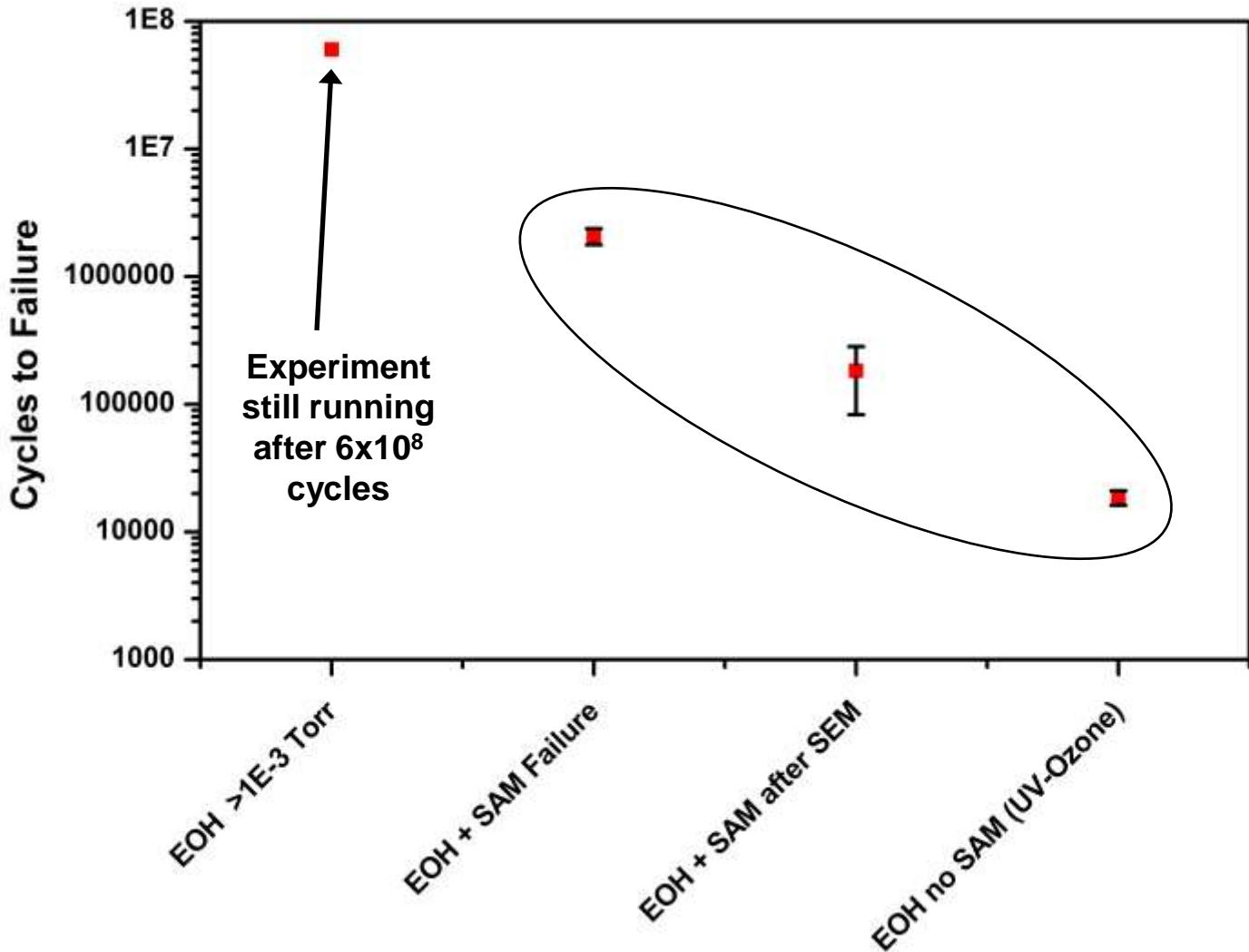
Brendan P. Miller and Jacqueline Krim\*

Sliding friction measurements of molecularly thin ethanol and pentanol films:  
How friction and spreading impact lubricity.

J. Low Temp. Phys. , Nov. 2009

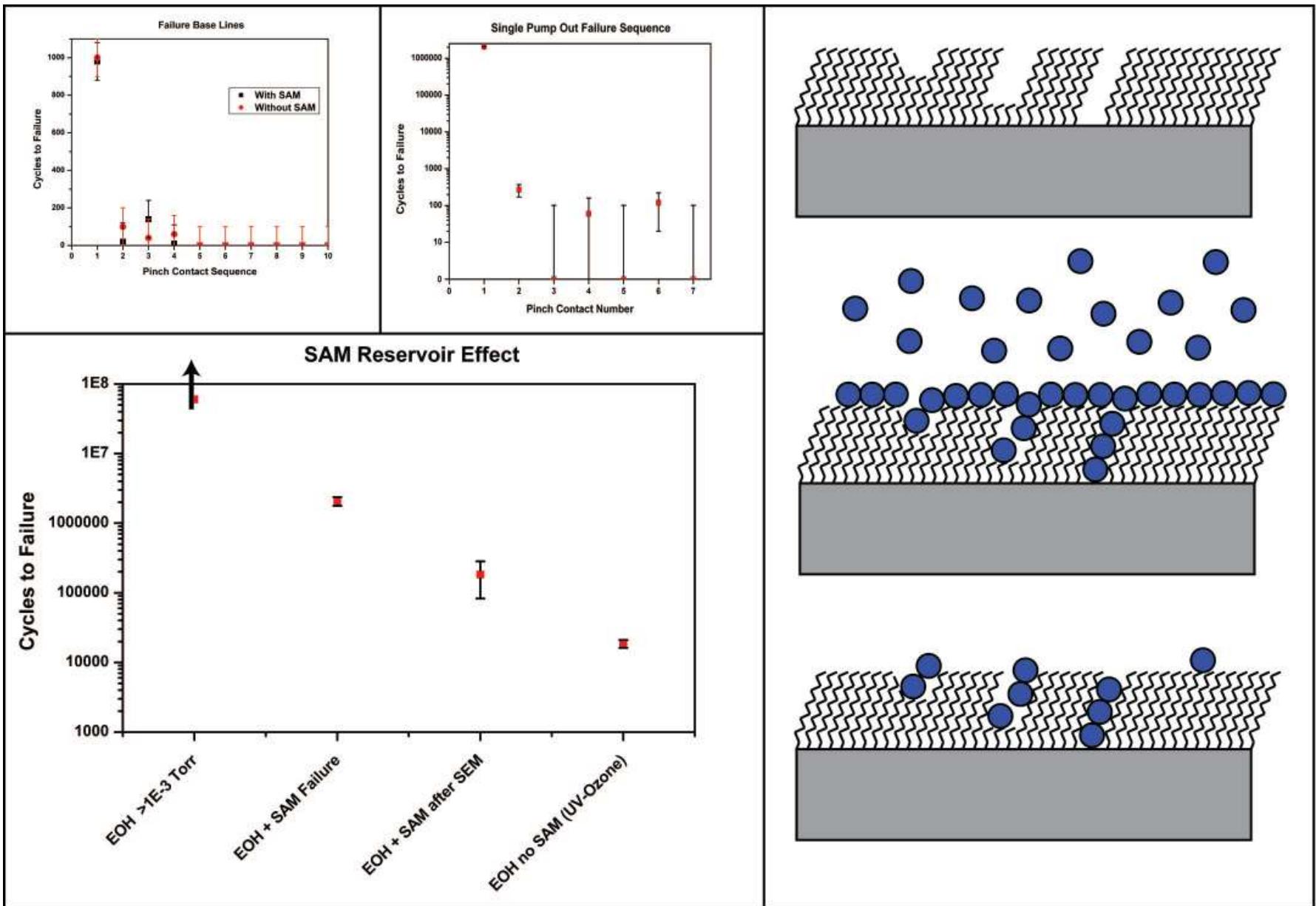


## SAM Reservoir Effect





# SAMS Reservoir effect for Ethanol





# Modeling Chemical Reactions in Materials Containing Carbon, Hydrogen & Oxygen: The qOAIREBO potential



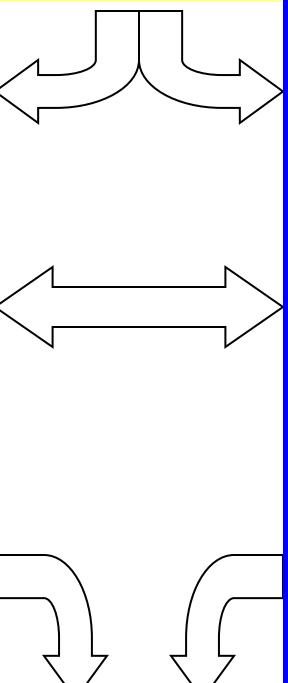
## Experimental Observation

Erdemir & Donnet, *J. Phys. D: Appl. Phys.* 39 (2006) R311. (a review of the friction of DLC)

The friction response of diamondlike carbon (DLC) and diamond is extremely sensitive to the presence of water vapor and other environmental conditions.

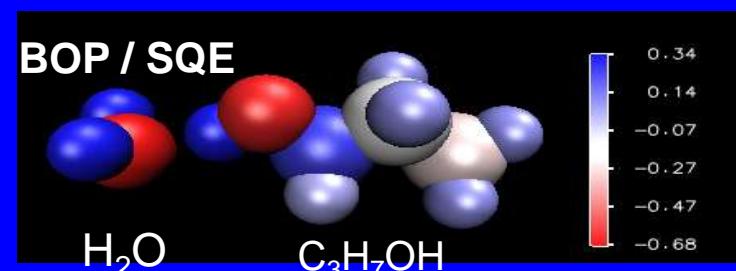
### Theory

- Existing methods to model fluctuating charges possess inherent flaws.
- Developed a method of integrating realistic charge fluctuations into ALL bond-order potentials (BOP/SQE). The method is scalable, transferable, & has none of the flaws identified in other methods.
- Chemical reactions in charge-containing systems can now be modeled.



### Simulation

- Atoms in small & large molecules have the correct charges (see below, colored by charge) DLC & water tribology can now be studied.



- Effects of alcohol VP lubricants & O termination can also be studied.

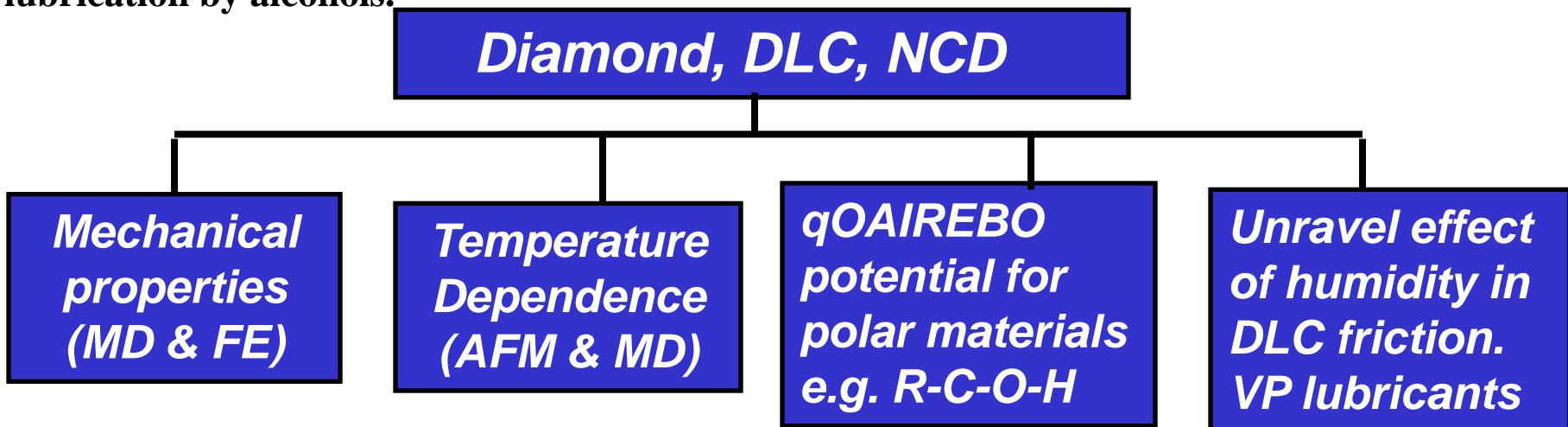
## Broader Impact

Our detailed studies of friction in DLCs will lead to moving MEMS components with enhanced lifetimes that can be deployed in a wide-range of environments.



# Modeling Water: Goals & Objectives

- **Goal:** Explain the fundamental mechanical & tribological behavior of diamondlike carbon (DLC) & related materials in different environments (e.g. humidity, temperature) using experiment, MD, & FE simulations. Develop reactive potential energy functions for diamond & DLC in the presence of water, which can also be used for vapor phase lubrication by alcohols.
- **Problem Statement:** No reactive potential energy function with fluctuating charges existed that could model water & DLC, or vapor phase lubrication with alcohols. How do the mechanical properties, adhesion, & tribology of diamond & DLC change with temperature & environment?
- **What we have learned:** Existing fluctuating charge models possess inherent flaws which render them useless in these applications. We have developed a simple way to correct these flaws and to model fluctuating charges & reactions for ALL bond-order formalisms. Our newly developed qOAIREBO can be used to model water & DLC and vapor phase lubrication by alcohols.





# Challenges and Unsolved Issues

## *From a fundamental viewpoint....*

- What are the underlying mechanisms that govern the friction of DLC & UNCD in the presence of water?
- Can we develop a “phase diagram” of tribological response for DLC ?
- Do the underlying mechanisms that govern friction in the presence of water play a role in other types of lubrication, such as, the vapor phase lubrication of MEMS by alcohols?

## *From a theory and modeling viewpoint....*

- How can the REBO potentials be altered to include 4 or more elemental types in one simulation? It's difficult to code 4 dimensional cubic splines for energy corrections ( $P_{ij}$ ).

## *From an applications viewpoint....*

- How do we turn this new basic knowledge into enhanced lifetimes and reliability for MEMS, spacecraft parts, jet engine bearings, pump seals, etc.



# Challenges and Unsolved Issues

## *From a fundamental viewpoint....*

- *What are the roles of electrons and phonons in transmitting frictional forces?*
- *What is the relationship between macroscopic and single asperity friction measurements?*

## *From a theory and modeling viewpoint....*

- *Construct a predictive theory of the temperature dependence of phononic friction.*

## *From an applications viewpoint....*

- *How do we turn this new basic knowledge into enhanced device performance?*



# Extreme Friction MURI (Highlight)

J. Krim PI-- (FA9550-041-0381)> --- North Carolina State University



**STATUS QUO**

- Silicon MEMS devices with rubbing contacts degraded within minutes.
- Focus on SAMS methods to prevent tribological failure.

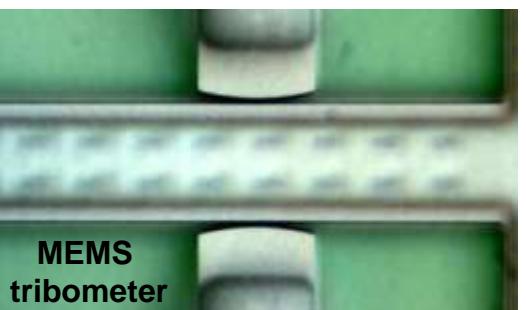
**NEW INSIGHTS**

- SAMS ruled out for protection against device failure in rubbing contact.
- Devices operational for indefinite durations in the presence of trace levels of alcohols. Atomic scale mechanisms proposed for multiple lubrication schemes.

**Silicon MEMS lubrication at ultra-low coverage**

## Main Achievements

- MEMS tribometer runs indefinitely with as little as  $1 \times 10^{-4}$  Torr of ethanol in chamber.
- When devices are dosed with ethanol to 1 monolayer coverage and then evacuated, SAM coatings allow devices to last orders of magnitude longer than those with damaged or destroyed SAMs
- Quartz Crystal Microbalance experiments confirm residual ethanol after vapor removal



**QUANTITATIVE IMPACT**

**Current Impact**  
Single and submonolayer lubrication is relevant far outside the nanometer device regime.

**Planned Impact**  
MEMS rubbing contact devices become viable, potentially including on-chip turbines for power generation



**END-OF-PHASE GOAL**

## Research Goals

A fundamental and atomistic understanding of how vapor phase lubrication solves the MEMS lubrication problem.